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Datasheet for the decision of 3 February 2017

Case Number: T 0132/12 - 3.3.05

Application Number: 04725504.7

Publication Number: 1608598

IPC: C03C3/06

Language of the proceedings: ΕN

Title of invention:

Silica glass containing TiO_2 , and process for its production

Patent Proprietor:

Asahi Glass Company, Limited

Opponent:

Corning Incorporated

Headword:

TiO2-SiO2 EUVL glass/ASAHI

Relevant legal provisions:

EPC Art. 83, 56

Keyword:

Sufficiency of disclosure - unusual parameter (no) Inventive step - unexpected improvement shown

Decisions cited:

T 0045/09

Catchword:



Beschwerdekammern Boards of Appeal Chambres de recours

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Case Number: T 0132/12 - 3.3.05

D E C I S I O N

of Technical Board of Appeal 3.3.05

of 3 February 2017

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Decision under appeal: Interlocutory decision of the Opposition

Division of the European Patent Office posted on 16 November 2011 maintaining European patent

No. 1608598 in amended form.

Composition of the Board:

Chairman E. Bendl

Members: J.-M. Schwaller

P. Guntz

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Summary of Facts and Submissions

- I. The present appeal lies from the interlocutory decision of the opposition division to maintain European patent No. 1 608 598 in amended form on the basis of the auxiliary request filed during the oral proceedings of 13 October 2011, with the independent claims reading as follows:
 - "1. A silica glass containing TiO_2 , which has a fictive temperature of at most 1,200°C, an OH group concentration of at most 600 ppm and a coefficient of thermal expansion of 0 \pm 200 ppb/°C from 0 to 100°C."
 - "2. A silica glass containing TiO_2 , which has a fictive temperature of at most 1,100°C, an OH group concentration of at most 600 ppm and a coefficient of thermal expansion of 0 \pm 200 ppb/°C from -50 to 150°C."
 - "6. A process for producing a silica glass containing TiO_2 as defined in any one of claims 1 to 5, which comprises:
 - (a) a step of forming a porous glass body by depositing and growing on a target quartz glass particles obtained by flame hydrolysis of a Si precursor and a Ti precursor as glass-forming materials;
 - (b) a step of heating the porous glass body to a vitrification temperature to obtain a vitrified glass body;
 - (c) a step of heating the vitrified glass body to a temperature of at least the softening temperature and forming it in a desired shape to obtain a formed glass body; and
 - (d) a step of carrying out annealing treatment wherein the formed glass body is held at a temperature exceeding 500°C for a predetermined time, and then, the

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temperature is lowered to $500\,^{\circ}\text{C}$ at an average cooling rate of at most $10\,^{\circ}\text{C/hr."}$

Claims 3 to 5 are dependent on claim(s) 1 and/or 2 and refer to preferred embodiments thereof.

- II. With its grounds of appeal, the opponent (hereinafter "the appellant") contested the impugned decision and requested *inter alia* that the patent be revoked on the grounds that it did not meet the requirements of Articles 83, 54 and 56 EPC.
- III. With its response to the grounds of appeal, the respondent filed six sets of claims as auxiliary requests 1 to 6 along with ten new documents, among them:
 - E1: D.-L. Kim, M. Tomozawa, "Fictive temperature of silica glass optical fibers re-examination", J. Non-Cryst. Solids, 286 (2001), pages 132-138
 - **E2:** H. Kakiuchida et al., "Precise determination of fictive temperature of silica glass by infrared absorption spectrum", J. Appl. Phys., 93 (2003), pages 777-779
 - E3: A. Agarwal et al., "A simple IR spectroscopic method for determining fictive temperature of silica glasses", J. Non-Cryst. Solids, 185 (1995), pages 191-198

E4: EP 1 152 237 A2

E5: EP 1 187 170 A2

E6: JP 09 241030 A and its partial English translation

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E7: EP 0 835 848 A2

IV. In a communication in preparation for oral proceedings, the board expressed its preliminary opinion that the claimed invention appeared to meet the requirements of Articles 83 and 54 EPC. The board further held document

D5: Corning Bulletin "ULETM Corning Code 7971 Titanium Silicate Zero Expansion Material" (1990); and Corning bulletin "Zero Expansion Glass ULETM" (1996)

to be the closest prior art, but left open the question whether or not the claimed subject-matter was obvious in particular from:

D10: WO 02/088036 A1, or

D11: WO 02/088035 A1.

V. The appellant responded that the subject-matter of claim 1 at issue was obvious vis-à-vis D5 or D11, which could both represent the closest prior art. The latter disclosure was in particular referred to in combination with document

D3: P. Schutz et al.: "Ultra-low expansion glasses and their structure in the SiO_2 -TiO₂ System", Amorphous Materials, pages 111 to 119 (1972).

VI. At the oral proceedings, which took place on 3 February 2017, the appellant announced that it waived its novelty objection. The discussion then focused on inventive step of the set of claims as maintained by the opposition division. The appellant argued that said claims lacked inventive step in the light of the disclosure of D5 taken in combination with D3, D11 or

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D9: EP 0 401 845 A1.

Regarding sufficiency of disclosure, the appellant said it maintained its objection but did not provide any further argument.

VII. At the end of the oral proceedings, the parties' requests were as follows:

The appellant requested that the decision be set aside and that the patent be revoked.

The respondent requested that the appeal be dismissed or that the patent be maintained in amended form on the basis of one of the sets of claims according to auxiliary requests 1 to 6 filed with the letter dated 8 October 2012.

VIII. The arguments of the parties, insofar as they are relevant to the present decision, can be summarised as follows:

The appellant held the fictive temperature to be an unusual parameter which did not enable the skilled person to perform the invention without undue burden. Regarding inventive step, starting from D5 as the closest prior art, the problem to be solved was merely the provision of an alternative, which was obvious to the skilled person.

For the respondent, the documents presented with the reply to the grounds of appeal showed that the parameter at issue, i.e. the fictive temperature, was commonly known before the priority date of the contested patent. Regarding inventive step, the problem was the one identified in paragraphs [0016] and [0064]

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of the patent specification. The proposed solution was not obvious, as already concluded by the department of first instance.

Reasons for the Decision

- 1. Sufficiency of disclosure
- 1.1 It is established case law that the requirements for sufficiency of disclosure under Article 83 EPC are met
 - (a) if, at the date of the application, the claimed invention could be performed by a person skilled in the art over the entire range claimed without undue burden, using common general knowledge and having regard to the information given in the patent in suit;
 - (b) if, where the definition of the claimed invention moreover includes one or more parameters, the skilled person can obtain sufficient information from the patent to verify whether the requirements concerning the parameters at issue are complied with (see e.g. decision T 0045/09, point 1.2).
- 1.2 In the present case, the claimed invention relates to a glass defined by several parameters, one of which the "fictive temperature" is considered by the appellant to be an unusual parameter which prevented the skilled person from carrying out the invention without undue burden.
- 1.3 As regards condition (a), the board observes that paragraphs [0040] to [0050] and the examples of the patent specification disclose ample details regarding the production of the claimed glass. Regarding the

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control of the fictive temperature, paragraph [0048] discloses that this is carried out by annealing the glass at a temperature exceeding 500°C, for example at a temperature of from 600 to 1,200°C, for at least 5 hours, and then lowering the temperature to not higher than 500°C at an average cooling rate of at most 10°C/hr.

In the present case the burden of proof lies with the opponent (the appellant) to show that a skilled person was unable to carry out the invention. In the absence of evidence to the contrary, it is credible that the above-mentioned production details disclosed in the patent in suit lead to a glass according to the invention.

- 1.4 With respect to condition (b), the board is not convinced that the fictive temperature is an unusual parameter which does not enable the skilled person to perform the invention without undue burden, because at least documents E1 to E7 clearly show that this parameter and the methods of measuring it were commonly known before the priority date of the contested patent, even at temperatures above 1,200°C.
- 1.5 That the different methods for measuring this parameter do not necessarily lead to the same values has no bearing on Article 83 EPC, since this difference does not hinder the skilled person from reproducing the invention as detailed in paragraphs [0040] to [0050] and in the examples of the patent.
- 1.6 The above arguments were in essence already set out in the communication stating the board's preliminary opinion and have not been contested by the appellant thereafter.

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- 1.7 It follows from the above that the board is not convinced that the claimed invention has not been disclosed clearly and completely enough to be carried out by a person skilled in the art. In other words, the requirements of Article 83 EPC are met.
- 2. Main request novelty

This issue being no longer contested, the board maintains the position expressed in its preliminary opinion, namely that the claimed subject-matter is not anticipated by the teaching of D11, which discloses a silica-titania glass having a low CTE variation (0 \pm 5 ppb/°C) and an OH content below 1 ppm, but which does not describe the fictive temperature of the glass as being at most 1,200°C or 1,100°C.

The subject-matter of all claims of the main request is therefore considered to meet the requirement for novelty under Article 54(1) and (2) EPC.

3. Main request - inventive step

Applying the problem-solution approach, the board came to the conclusion that the set of claims as maintained by the opposition division involves an inventive step for the following reasons:

3.1 Document D5, which the board considers and the parties acknowledged as the closest prior art, discloses a titanium silicate glass having a coefficient of thermal expansion (CTE) which falls within the range disclosed in claim 1 (see D5, page 3, figure "instantaneous CTE"), a fictive temperature between 877 and 888°C and an OH group concentration between 825 and 843 ppm (see D17: affidavit of W.R. Rosch, appendix 1).

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The subject-matter of claim 1 thus differs from the above disclosure in that the OH group concentration of the glass is lower, namely at most 600 ppm. This was common ground between the parties.

- 3.2 According to the patent in suit (paragraphs [0016] and [0064]), the problem underlying the invention is to provide a TiO_2 -containing silica glass having a CTE of substantially zero over a wide temperature range, a small fictive temperature fluctuation and a more uniform CTE.
- 3.3 As the solution to this problem, the contested patent proposes the subject-matter of claim 1 at issue, which is in particular characterised in that the OH group concentration is at most 600 ppm.
- 3.4 As to whether the problem identified in point 3.2 has been solved, the board notes that the glass of D5 has a CTE of substantially zero (see the lower graph on page 4 of D5) over a wide temperature range (see point 3.1 above), and so this part of the problem is manifestly already solved.
- 3.4.1 The board however does not share the appellant's view that the problem is therefore to be reformulated as the provision of an alternative titania-silica glass. As shown by Table 2 of the patent, the glass according to the claimed invention, as represented by example 5 which has an OH concentration of 70 ppm has a smaller fictive temperature fluctuation than the glass of example 6, which represents the glass according to D5 (ULETM).

Furthermore, as explained by the respondent, the smaller fluctuation of fictive temperature of the

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claimed glass is closely correlated with a more uniform CTE in comparison to D5, because the fluctuation of fictive temperature corresponds to the difference between:

- the fictive temperature measured after rapid cooling in air of a piece of glass annealed at 900°C for 100 hours, and
- the fictive temperature measured after slowly cooling a similarly annealed piece of glass at a rate of 5°C/hr ,

which, as mentioned above, is shown in examples 5 and 6 of the patent in suit.

Furthermore, this rapid and slow cooling of the glass samples can be regarded as a simulation of the cooling of the surface and the core of the glass block. The measurement of the fictive temperature of these differently treated samples 5 and 6 therefore represents the measurement of the fictive temperatures on the surface and in the core of the glass block. The smaller the measured difference, the more uniform the CTE of the glass block.

3.4.2 The appellant contested these results, without however providing any evidence for its assertions.

In the absence of evidence to the contrary and on the basis of the examples disclosed, it is thus credible - as confirmed by paragraph [0064] of the patent - that the present invention provides for a more uniform CTE of the glass.

3.4.3 The appellant further disputed that the above effect was obtained over the whole breadth of the claims, in particular when the glass had an OH content close to

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600 ppm, again however without providing any evidence for its assertions.

- 3.4.4 It follows from the above that the problem identified in point 3.2 is held to be partly solved since an improvement in terms of uniformity of CTE and fictive-temperature fluctuation has been proven. The problem can therefore be reformulated as the provision of a TiO2-containing silica glass having a small fictive temperature fluctuation and a more uniform CTE. In view of example 5 of the patent in suit the board has no doubt that the solution proposed has successfully solved the problem over the entire range claimed.
- 3.5 To assess the inventiveness of the claimed subjectmatter, when starting from D5 as discussed above, it
 has to be determined whether the proposed solution was
 obvious in the light of the prior art, in particular
 documents D3, D9 or D11 that the appellant held to
 disclose the above solution.
- 3.5.1 For the board, the solution is not obvious, because none of these documents teaches reducing the OH concentration of a titania-silica glass for the purpose underlying the invention, namely to improve the uniformity of the CTE and lower the fluctuation of the fictive temperature in a titania-silica glass.
- 3.5.2 Document D3 in particular is totally silent regarding this particular effect and with respect to the OH content.
- 3.5.3 D9 (paragraph [0020]) concerns the production of optical blanks with improved resistance to deterioration upon exposure to a high-power UV laser beam. This is solved e.g. by providing more uniform

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optical properties (paragraph [0024]), in particular a highly homogeneous refractive index distribution (paragraph [0025]), which effect is obtained in particular by making the fictive temperature of the glass uniform (see paragraph [0017]). The optical blanks are made from silica glass containing at least 50 ppm of OH groups ([0032]).

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For the board, the skilled person would not consider this document because it does not concern the ${\rm TiO_2}-$ containing silica glass of the invention, but high-purity silica glasses which, as explained in D9, paragraph [0046], should not contain more than 50 ppb in total of Ti, Cr, Fe, Ni and Cu, and preferably not more than 10 ppb Ti.

Furthermore, D9 is concerned with a different problem, namely providing more uniform optical properties, in particular a highly homogeneous refractive index distribution, which a priori has nothing in common with the purpose of the invention, namely achieving a more uniform CTE.

It follows from the above considerations that even if the skilled person had an incentive to consult D9, he would not arrive at the wording of claim 1 at issue.

3.5.4 D11 is concerned with the same type of glasses as those underlying the invention, namely TiO₂-containing silica glass with a very low OH content (preferably less than 1 ppm in claim 4) and low CTE variations. However - as explained in item 2 above - D11 does not describe any fictive temperature, let alone one of at most 1,200°C or 1,100°C.

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Furthermore, D11 is not concerned with the purpose of the invention, namely achieving a more uniform CTE but rather with the problem of rendering a high-purity silica-glass tolerant to infrared transmission or deep-UV applications (see D11, page 1, lines 7 to 9 or page 2, lines 7 to 9). It follows that the skilled person would again not arrive at the claimed subject-matter from the above teachings.

- 3.5.5 For the board, starting from document D5, the other documents in the proceedings neither disclose nor suggest the solution as defined in claim 1 at issue to solve the problem underlying the invention.
- 3.6 Accordingly, it follows from the above considerations that, having regard to the state of the art, the subject-matter of claim 1 at issue is not obvious to a person skilled in the art, and so meets the requirements of Article 56 EPC.
- 3.7 The same conclusion applies to independent claims 2 and 6, and claims 3 to 5, which depend on claim(s) 1 and/or 2.
- 4. As the appellant has not succeeded in showing that the set of claims as maintained by the opposition division does not meet the requirements of the EPC, its appeal fails.

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Order

For these reasons it is decided that:

The appeal is dismissed

The Registrar:

The Chairman:



C. Vodz E. Bendl

Decision electronically authenticated